Article:

Vopsaroiu, M, Thwaites, M J, Rand, S et al. (2 more authors) (2004) Novel sputtering-technology for grain-size control. IEEE Transactions on Magnetics. pp. 2443-2445. ISSN 1941-0069

https://doi.org/10.1109/TMAG.2004.828971

Novel Sputtering Technology for Grain-Size Control

Marian Vopsaroiu, M. J. Thwaites, S. Rand, P. J. Grundy, and K. O'Grady, *Member, IEEE*

***Abstract*—In this paper, we present a description of a novel high-rate plasma sputtering system that allows the control of grain size in sputtered films. Additionally, the system has the advantage of a better utilization of the target material (around 80% to 90%) by eliminating the race track at the target as in conventional plasma magnetron sputtering systems. The potential and capabilities of this novel plasma sputtering device are demonstrated in this paper by the deposition of a number of different Cr thin films suitable for underlayers in thin-film media and for which we have performed a systematic X-ray and TEM analysis to determine the grain-size histograms, mean grain diameters, and their relationship to the sputtering processes.**

***Index Terms*—Cr thin films, grain-size control, novel plasma sputtering.**

I. Introduction

The ability to sputter thin films with controlled grain-size distribution and texture is essential for numerous industrial applications, especially in magnetic recording industry. In this paper, we describe a novel plasma sputtering system that has considerable advantages, including the ability to control the average grain size in sputtered films without the use of seed layers. A number of Cr thin films have been sputtered using different process conditions in order to determine the optimum sputtering process parameters in terms of average diameter, standard deviation, and crystallographic orientation of the Cr grains. Cr and Cr alloys are technologically important in the magnetic recording industry, where they are used as underlayers for CoCrPt [1] or CoCrTa [2] longitudinal magnetic recording media. The crystallographic orientation and the grain size in the Cr underlayer promotes the epitaxial growth of the magnetic layer [3]. Achieving higher recording densities is possible by growing smaller grains with a more uniform grain-size distribution. However, thermal decay of the signal in high density media requires grains with high magnetic anisotropy and narrow-size distribution, so that the particles are also thermally stable and the medium has a better signal-to-noise ratio. Consequently, controlling the average grain size and the grain-size distribution is very important for achieving high-density and thermally-stable media. The objectives of the present study are to investigate the grain-size evolution as a function of the sputtering process parameters using a novel plasma sputtering device that is described in Section II.

Manuscript received October 16, 2003. This work was supported in part by the U.K. Department for Trade and Industry (DTI) and in part by the U.K. Department for Trade and Industry, LINK Information Storage and Displays Programme.

M. Vopsaroiu and K. O'Grady are with The University of York, Magnetic Materials Research Group, Department of Physics, York, YO10 5DD, U.K. (e-mail: mv4@york.ac.uk; kog1@york.ac.uk).

M. J. Thwaites and S. Rand are with Plasma Quest, Ltd., Hook, RG27 9UT, U.K. (e-mail: mike.thwaites@plasmaquest.co.uk; stuart.rand@plasmaquest.co.uk).

P. Grundy is with The University of Salford, Salford, M5 4WT, U.K. (e-mail: p.j.grundy@salford.ac.uk).

Digital Object Identifier 10.1109/TMAG.2004.828971

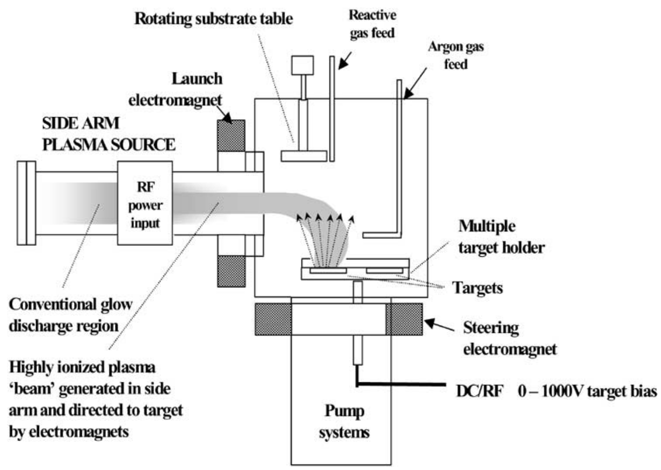

Fig. 1. Schematic diagram of HiTUS plasma sputtering system at the University of York. By changing the polarity of the steering electromagnet, the plasma beam can be directed to the substrates for intentional heating or plasma cleaning of the substrates.

II. Description of the High Target Utilization Plasma Sputtering (HiTUS)

Our novel plasma sputtering system is designed so that the plasma is produced by means of a 2.5 kW RF antenna in a side arm, remote from the sputtering chamber (Fig. 1). The plasma beam is then guided to the water-cooled target through the use of magnetic fields, resulting in a high-density plasma at the target surface. This is coupled with the DC/RF bias voltage applied to the target and allows highly efficient and controllable sputtering of the target material. The separation of the plasma generation from the target and the sputtering chamber is the key element in achieving a wide control of the process parameters, thereby allowing optimum deposition conditions to be established for a given application [4].

Unlike conventional magnetron sputtering in which race-tracks are formed on the target, this novel system eliminates the need of magnetic fields at the target and uniform sputtering is produced. Consequently, the target utilization is improved from about 25% in conventional magnetron sputtering to 80–90% in this system, hence the name high target utilization sputtering (HiTUS) [4]. HiTUS technology also allows sputtering from thick ferromagnetic targets and the use of mosaic targets for the preparation of alloys with varying compositions.

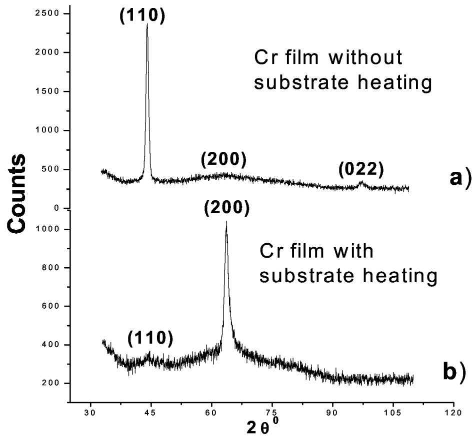

Fig. 2. X-ray spectra of a Cr film sputtered with and without plasma substrate heating.

III. Results and Disscusions

Three different sets of Cr thin films have been sputtered using HiTUS. A multiple substrate holder allowed us to sputter up to six samples in a single run without breaking vacuum. Prior to each sputtering process, plasma cleaning has been employed at both the target (60 s) and substrate (100 s) in order to eliminate possible contamination or oxide layers formed at the material/air interface. All Cr films were sputtered onto glass substrates after pumping to a base pressure of about 7×10^{-7} mbar. Depending on the sputtering conditions, typical growth rates are between 10 to 30 nm/min. During sputtering, the substrate temperature was about 100 °C due to plasma heating. X-ray diffraction showed a consistently dominant (110) crystallographic orientation of the samples sputtered without intentional substrate heating. However, for the growth of Co alloys with the c axis in-plane, it is well known that a (200) preferred orientation in the Cr underlayer is required. This is obtained by heating the substrate to >250 °C [5]. Using pre-heating of substrates by exposure to the plasma beam for up to 3 min, we have achieved the change from (110) to (200) orientation in our Cr films (Fig. 2). In order to study the control of the grain size as a function of the sputtering parameters, three sets of Cr samples were sputtered at constant substrate and target temperatures as follows:

Set A) Ar process pressure 2.2×10^{-3} mbar, bias voltage at the target $= -800$ V. RF power varied from 0.62 to 2.25 kW.

Set B) Ar process pressure 2.2×10^{-3} mbar, RF power $=$ 1.75 kW. Bias voltage at the target varied from -500 to -1000 V.

Set C) Bias voltage at the target $= -800$ V, RF power $=$ 1.75 kW. Ar process pressure varied from 1.19 to 4.7×10^{-3} mbar.

In this preliminary work, the film thickness could not be measured for each sample, but it was typically around 50 nm.

Carbon-coated TEM grids were attached to the glass substrates and TEM images acquired for each sample. Over 500 particles for each sample were measured as indicated in [6]. Plane-view TEM images were obtained in bright field mode at 120 kV and x 150k magnification. Figs. 3(a) and 4(a) show two TEM images corresponding to samples from set A having the smallest and the largest average grain size, respectively. The mean grain size has been obtained for all samples by measuring and counting the grain diameters with a Zeiss particle-size analyzer. Typical size distribution histograms corresponding to the images in Figs. 3(a) and 4(a) are displayed below each image [see Figs. 3(b) and 4(b), respectively], showing a log-normal

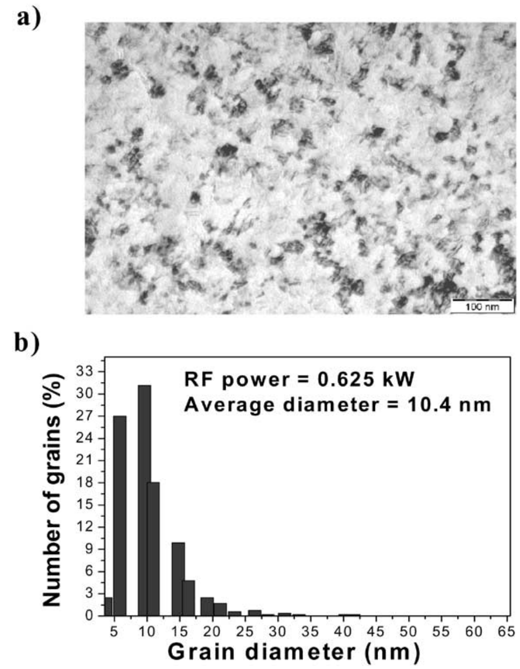

Fig. 3. (a) Typical TEM image. (b) The corresponding particle-size distribution measured for a sample of set A sputtered at 0.62 kW RF power.

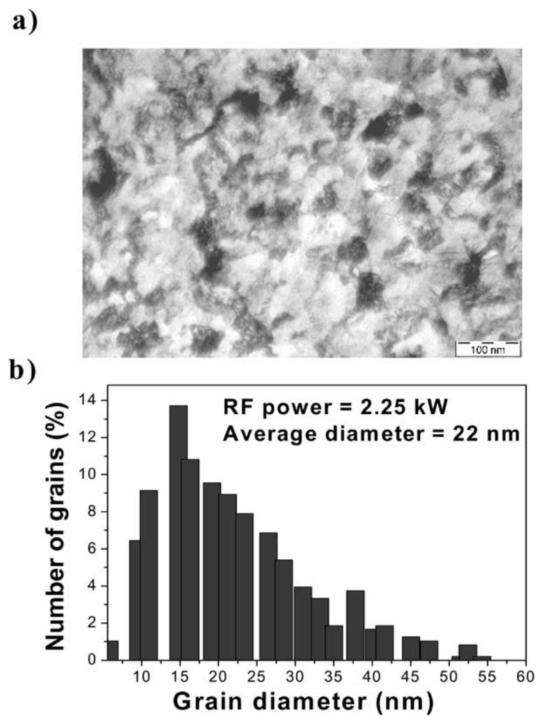

Fig. 4. (a) Typical TEM image. (b) The corresponding particle-size distribution measured for a sample of set A sputtered at 2.25 kW RF power.

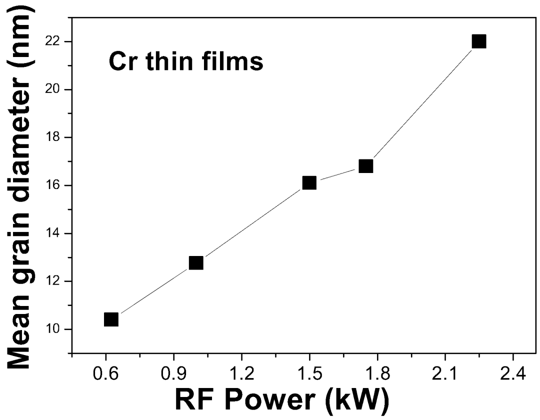

Fig. 5. Mean grain diameter as a function of the RF power.

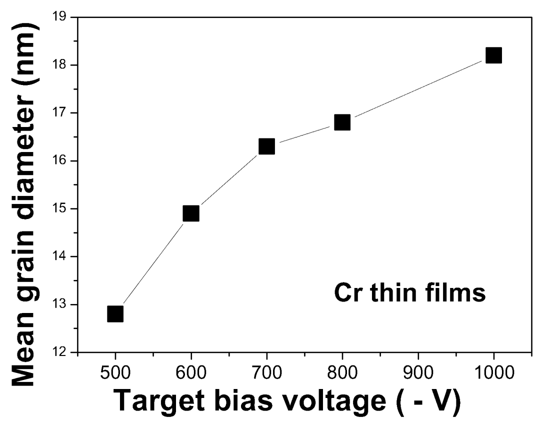

Fig. 6. Mean grain diameter as a function of the bias voltage at the target.

type distribution [6]. The widths of the distribution, as well as the mean grain sizes vary, with sputtering conditions. The mean grain size for both sets of samples have been determined using a standard statistical averaging while the full fitting of the distributions and the standard deviation analysis will be presented elsewhere. There is a clear variation of the mean grain size with sputtering conditions as indicated in Figs. 5–7, where each diagram represents the mean grain diameter variation as a function of the RF power, bias voltage, and process pressure, respectively.

IV. Conclusion

We have presented details of a novel plasma sputtering system (HiTUS) that has the capability to produce thin films with controlled grain size and texture. The system was initially tested on

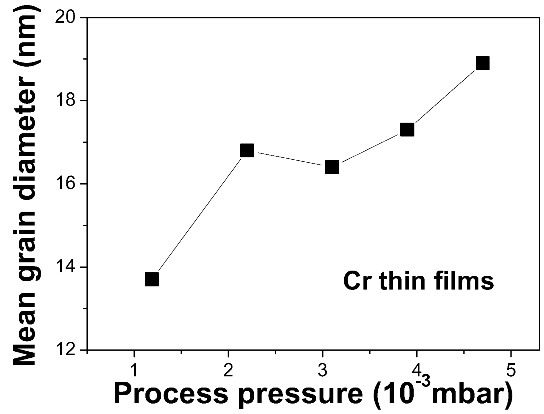

Fig. 7. Mean grain diameter as a function of the process pressure.

Cr thin films and showed a controllable variation of the mean grain size with sputtering process parameters (see Figs. 5–7). Although the physical mechanisms leading to a grain-size control in this complex plasma sputtering system are not fully understood, we conclude that a faster sputtering rate will generate a bigger mean grain diameter. This is supported by the results since either a higher RF power, higher bias voltage, or higher process pressure will generate a faster sputtering rate. A possible explanation could be that the variation in grain size for samples prepared in different sputtering conditions can be related to the crystal symmetry in which a higher symmetry means a higher probability for two neighboring grains to have closer lattice orientations and, therefore, to join together forming a bigger grain during the growth process [7]. This is also supported by the X-ray data for our samples, which showed a better crystallographic orientation for samples with higher grain diameters.

References

[1] S. C. Oh and T. D. Lee, "A study on VMn underlayer in CoCrPt longitudinal media," *IEEE Trans. Magn.*, vol. 37, pp. 1504–1507, July 2001.

[2] H. Jia, J. Veldeman, and M. Burgelman, "Magnetic properties and microstructure of CoCr and CoCrTa thin films sputtered at high pressure on a PET substrate," *J. Magn. Magn. Mater.*, vol. 223, pp. 73–80, 2001.

[3] J. K. Park, J. H. Kim, S. I. Park, H. M. Lee, and D. Y. Oh, "Evolution of grain structure of as-deposited Cr thin films with deposition temperature," *Script. Mater.*, vol. 48, pp. 1161–1166, 2003.

[4] M. J. Thwaites, "High Density Plasmas," U.S. Patent 6 463 873, Oct. 15, 2002.

[5] B. Y. Wong and D. E. Laughlin, "Controlling the magnetic properties of CoCrPt thin films by means of thin hexagonal-close-packed intermediate layers," *Appl. Phys. Lett.*, vol. 61, pp. 2533–2533, 1992.

[6] G. R. Jones, M. Jackson, and K. O'Grady, "Determination of the grain size in thin films," *J. Magn. Magn. Mater.*, vol. 193, pp. 75–78, 1999.

[7] B. Valcu, Y. Takahashi, and H. N. Bertram, "Effect of crystallographic orientation on grain size distribution," *J. Appl. Phys.*, vol. 92, no. 10, pp. 6094–6098, 2002.